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[54] Title of Invention:

Fluidized Bed Catalyst for Propylene or Isobutylene

Ammoxidation

[57] [Abstract]

A fluidized bed catalyst for the production of acrylonitrile or methacrylonitrile by propylene or isobutylene ammoxidation. The active components comprised by the catalyst used are expressed by the general formula $Mo_{(12-g)}Bi_aFe_bNi_cX_dY_eZ_fQ_gO_x$, wherein X is one or a mixture of two or more elements from among Mg, Co, Ca, Be, Cu, Zn, Pb, Sb, Mn, Cr; Y is one or a mixture of two or more elements from among La, Ce, Sm; Z is one or a mixture of two or more elements from among K, Rb, Na, Cs; Q is one or a mixture of two or more elements from among B, W, P, V; a ~ f are the numbers of atoms of appropriate elements and x is the sum total of oxygen atoms required by the valences of the other elements. Compared to the existing process, this catalyst enables the conducting of reaction under low oxygen ratio and ammonia ratio and lower temperatures; furthermore, it has a higher acrylonitrile yield.

SCOPE OF PATENT CLAIMS

1. A fluidized bed catalyst for the production of acrylonitrile or methacrylonitrile by propylene or isobutylene ammoxidation which comprises Mo, Bi, Fe, Ni and, optionally, one or more oxides of metals from among Mg, Co, Ca, Be, Cu, Zn, Pb, Sb, Mn, Cr, La, Ce, Sm, K, Rb, Na, Cs, B, W, P or V and the active ingredients contained therein are expressed by the general formula:

$$Mo_{(12-g)}Bi_aFe_bNi_cX_dY_eZ_fQ_gO_x$$

in which formula:

X is one or a mixture of two or more elements from among Mg, Co, Ca, BeCu, ZN, Pb, Sb, Mn, Cr;

Y is one or a mixture of two or more elements from among La, Ce, Sm;

Z is one or a mixture of two or more elements from among K, Rb, Na, Cs;

Q is one or a mixture of two or more elements from among B, W, P, V;

 $a = 0.1 \sim 6$, b, c, d, e, $g = 0.1 \sim 10$, $f = > 0 \sim 0.5$, x is the sum total of oxygen atoms required by the valences of the other elements.

The support utilized by the catalyst is silicon dioxide or alumina or a mixture of the two, and its content is $(40 \sim 60)$ % (wt.) of the total amount of catalyst.

- 2. A method of manufacturing acrylonitrile or methacrylonitrile described in Claim 1 characterized in that X in the above-mentioned catalyst is Mg or Co or a mixture thereof.
- 3. A method of manufacturing acrylonitrile or methacrylonitrile described in Claim 1 characterized in that Y in the above-mentioned catalyst is La or Ce or a mixture thereof.
- 4. A method of manufacturing acrylonitrile or methacrylonitrile described in Claim 1 characterized in that Z in the above-mentioned catalyst is Rb or Cs or a mixture thereof.
- 5. A method of manufacturing acrylonitrile or methacrylonitrile described in Claim 1 characterized in that Q in the above-mentioned catalyst is W or V or a mixture thereof.
- 6. A method of manufacturing acrylonitrile or methacrylonitrile described in Claims 1, 2, 3 or 4 characterized in that the support of the above-mentioned catalyst is silicon dioxide.

FLUIDIZED BED CATALYST FOR PROPYLENE OR ISOBUTYLENE AMMOXIDATION

This invention pertains to a catalyst for manufacturing unsaturated nitriles by olefin ammoxidation, wherein the active ingredients of the catalyst used are molybdenum, bismuth, iron, an a variety of metal oxides.

Mo-Bi catalysts are well known and commonly used in the reaction that produces unsaturated nitriles from olefins (such as producing acrylonitrile or methacrylonitrile from propylene or isobutylene). Moreover, ever since the Mo-Bi catalysts first appeared, improvements to their compositions have been continuously patented. For example, adding transition metals to the active ingredients to increase activity and raise the product yield-per-pass; adding rare-earth elements to increase redox capacity; adding Na, P, and other elements to improve the catalyst's abrasion performance; adding Fe, Co, Ni and other elements to suppress Mo volatilization and increase catalyst stability etc. In recent years, the research of acrylonitrile catalysts has been focused on the catalyst's ability to efficiently utilize oxygen or ammonia, i.e. on enabling the conducting of a reaction with a lower oxygen ratio and ammonia ratio. It is obvious that a lower oxygen ratio and ammonia ratio will increase the yield, but lower ammonia ratio is even more important to reduce the unreacted ammonia content of the tail gas, and, accordingly, reduce the amount of acid used to neutralize unreacted ammonia. This is very advantageous for the reduction of production costs and pollution control. In patent CN 96101529.2 an improved Mo-Bi catalyst was introduced whose advantage is the better redox stability of the catalyst; thus the reaction can produce a better yield with lower oxygen ratio and ammonia ratio of the feed.

The goal of this invention is to offer a new Mo-Bi catalyst. By comparison to similar catalysts introduced in comparison documents, it retains their advantages while at the same time offering a higher propylene conversion and a higher acrylonitrile or methacrylonitrile yield-per-pass.

The catalyst offered in this invention comprises Mo, Bi, Fe, Ni and, optionally, one or more oxides of metals from among Mg, Co, Ca, Be, Cu, Zn, Pb, Sb, Mn, Cr, La, Ce, Sm, K, Rb, Na, Cs, B, W, P or V and the active ingredients contained therein are expressed by the general formula:

$$Mo_{(12\text{-g})}Bi_aFe_bNi_cX_dY_eZ_fQ_gO_x$$

in which formula:

X is one or a mixture of two or more elements from among Mg, Co, Ca, Be, Cu, Zn, Pb, Sb, Mn, Cr;

Y is one or a mixture of two or more elements from among La, Ce, Sm;

Z is one or a mixture of two or more elements from among K, Rb, Na, Cs;

Q is one or a mixture of two or more elements from among B, W, P, V;

 $a = 0.1 \sim 6$, b, c, d, e, $g = 0.1 \sim 10$, $f = >0 \sim 0.5$, x is the sum total of oxygen atoms required by the valences of the other elements.

The support utilized by the catalyst is silicon dioxide or alumina or a mixture of the two, and its content is $(40 \sim 60)$ % (wt.) of the total amount of catalyst.

However, the best X is Mg or Co or a mixture thereof; the best Y is La or Ce or a mixture thereof, the best Z is Rb or Cs or a mixture thereof, and the best Q is W or V or a mixture thereof. The preferred support is silicon dioxide.

The catalyst can be prepared by the slurry method known to those skilled in the art or by other universally known methods. The raw materials usually applied for catalyst components are nitrates or other water-soluble salts and acids, to facilitate the uniform mixing of components.

Compared to the existing processes, the catalyst offered in this invention has a better redox capacity and very good redox stability within a wider range of reaction conditions. Therefore a higher product yield-per-pass can be achieved with lower air / olefin ratio and lower ammonia / olefin ratio, and, presuming that the higher olefin conversion rate is preserved, acrylonitrile or methacrylonitrile selectivity is dramatically increased. Moreover, the catalyst offered in this invention has a comparatively lower reaction operation temperature, which has an extremely favorable effect on extending the catalyst service life.

Below this invention will be described in greater detail with Practical Examples. The positive effect of this invention is clearly demonstrated by the evaluation data of the Practical Examples.

In the Practical Examples below, the actual conditions of the catalyst examination and evaluation were as follows:

Reactor:

fluidized bed reactor, internal diameter 38 mm

Catalyst charge:

550 g

Pressure at the top of the reactor:

0.085 / 0.11 / 0.14 MPa (gauge)

Reactor temperature:

420 / 410°C

Reaction time:

4 hours

Raw material ratio:

propylene / ammonia /air = 1 / 1.15 / 9.5 (mole ratio)

WWH:

0.06

The reaction product was absorbed by acid liquor and water at 0° C. The product was analyzed by a combination of gas phase chromatography and chemical analysis. Carbon balance was calculated and significant data were those obtained when the carbon balance was $(95 \sim 105)\%$.

The definitions of propylene conversion rate, acrylonitrile yield and selectivity are as follows:

Practical Example 1

423.2 g of (NH₄)6Mo₇O₂₄·2H₂O was placed in 330 g of water at 70°C and stirred till it was completely dissolved. The 1250 g of 40% (wt) silicasol was added thereto to make substance A.

Fed propylene mole number

166 g of Fe(NO₃)₃·9H₂O were added to 40 g of hot water at 70°C and stirred until it was dissolved, then to it were added 91.3 g of Bi(NO₃)₃·5H₂O, 292.8 g of Ni(NO₃)₃·6H₂O, 104.5 g of Mg(NO₃)₃·6H₂O, and 46.7 g of Sm(NO₃)₃·nH₂O; upon stirring to dissolving substance B was obtained.

1.9 g of KNO₃ and 1.5 g of RbNO₃ were dissolved in 20 g of water and then added to substance B to form substance C.

Substance C was added drop by drop to substance A while quickly stirring and, after ageing at 70°C for 3 hours, it was spray dried, the resulting particles were calcined for 3 hours in a rotary calciner, and the catalyst product was obtained.

Practical Examples $2 \sim 13$

The method of catalyst preparation was the same as in Practical Example 1, except that the starting materials and their corresponding amounts were different.

The starting materials and their corresponding amounts used in Practical Examples 1 ~13 are listed in Table 1; the composition of the various active ingredients of individual catalysts is shown in Table 2.

The catalyst products were examined and evaluated based on the examination and evaluation conditions stated above, and the results were included in Table 3.

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[54]发明名称 丙烯、异丁烯氨氧化流化床催化剂 [57] 養基

本发明属于一种用于丙烯、异丁烯氢氧化制取丙烯 腈、甲基丙烯腈的的流化床 催化剂,含有的活性组份由 通式 Mo(12-x) Bi, Fe, Ni, X, Y, Z, Q, O, 表示, 其中 X 为 Mg、 Co、Ca、Be、Cu、Zn、Pb、Sb、Mn、Cr 中的一种或两种以上 的混合物:Y为La、Co、Sm中的一种或两种以上的混合 物;Z为 K、Rb、Na、Cs 中的一种或两种以 上的混合物:O 为 B、W、P、V 中的一种或两种以上的混合物;a~f 为相 应 元素的原子数,x 为满足其它元素化合价所需的氧原 子总数。与已有技术相比,本催化剂可以较低的氧比和 氨比以及较低的温度进行反应。且有较高的丙烯腈 收 率。

1、一种用于丙烯、异丁烯氨氧化制取丙烯腈、甲基丙烯腈的流化床催化剂,含有 Mo、Bi、Fe、Ni,以及任选地含有 Mg、Co、Ca、Be、Cu、Zn、Pb、Sb、Mn、Cr、La、Ce、Sm、K、Rb、Na、Cs、 B、W、P或 V 中的一种或多种金属氧化物,其含有的活性组份由下列通式表示:

 $Mo_{(12-g)} Bi_a Fe_b Ni_c X_d Y_e Z_f Q_g O_x$

其中:

X 为 Mg、Co、Ca、Be、Cu、Zn、Pb、Sb、Mn、Cr 中的一种或两种以上的混合物,

Y为 La、Ce、Sm 中的一种或两种以上的混合物,

Z为 K、Rb、Na、Cs 中的一种或两种以上的混合物,

Q为B、W、P、V中的一种或两种以上的混合物,

 $a = 0.1 \sim 6$, b、c、d、e、g = $0.1 \sim 10$, f = $> 0 \sim 0.5$, x 为满足其它元素化合价所需的氧原子总数。

催化剂所用的载体为二氧化硅或氧化铝或两者的混合物,其含量为催化剂总量的(40~60)%(w.t.)。

- 2、根据权利要求 1 所述的催化剂, 其特征在于 X 为 Mg 或 Co 或其混合物。
- 3、根据权利要求 1 所述的催化剂, 其特征在于 Y 为 La 或 Ce 或其混合物。
- 4、根据权利要求 1 所述的催化剂, 其特征在于 Z 为 Rb 或 Cs 或其混合物。
 - 5、根据权利要求 1 所述的催化剂, 其特征在于 Q 为 W 或 V 或其

混合物。

6、根据权利要求 1、2、3 或 4 所述的催化剂, 其特征在于所用的载体为二氧化硅。

说

丙烯、异丁烯氨氧化流化床催化剂

本发明涉及由烯烃氨氧化制取不饱和腈的催化剂,其活性组份含有 钼、铋、铁等多种金属氧化物。

Mo-Bi 催化剂是用于烯烃制取不饱和腈(如由丙烯、异丁烯氨氧化 制取丙烯腈或甲基丙烯腈)的反应中较成熟和较常用的催化剂,且自诞 生以来不断有专利推出新的改进配方。如在活性组份中加入过渡金属以 提高活性,增加产物的单收;加入稀土元素改善氧化还原能力;加入 Na、 P 等元素以提高催化剂的耐磨性; 加入 Fe、Co、Ni 等元素以抑制 Mo 的 挥发,提高催化剂的稳定性等等。而近年来对丙烯腈催化剂的研究已较 多地注重催化剂能够高效地利用氧或氨,即可以较低的氧比和氨比进行 反应。显然,较低的氧比和氨比使得收率提高,然而低氨比具有更重要 的意义是降低了反应尾气中未反应氨的含量,从而可相应减少用于中和 未反应氨的酸的用量,这对降低生产成本和环境污染的控制都是极为有 利的。专利 CN 96101529.2 介绍了一种改进的 Mo-Bi 催化剂,其优点是 催化剂具有较好的氧化还原稳定性、反应可以较低的氧比和氨比进料而 仍有较好的反应收率。

本发明的目的是提供一种新的 Mo-Bi 催化剂, 与上述对比文献介绍 的同类催化剂相比,除仍具备其拥有的优点外,同时还有相对更高的丙 烯转化率和较髙的丙烯腈或甲基丙烯腈单程收率。

本发明提供的 Mo-Bi 催化剂含有 Mo、Bi、Fe、Ni,以及任选地含 有 Mg、Co、Ca、Be、Cu、Zn、Pb、Sb、Mn、Cr、La、Ce、Sm、K、

Rb、Na、Cs、 B、W、P或V中的一种或多种金属氧化物,其含有的活性组份由下列通式表示:

Mo (12-g) Bi_a Fe_b Ni_c X_d Y_e Z_f Q_g O_x

其中:

X为 Mg、Co、Ca、Be、Cu、Zn、Pb、Sb、Mn、Cr 中的一种或两种以上的混合物,

Y为 La、Ce、Sm 中的一种或两种以上的混合物,

Z为 K、Rb、Na、Cs 中的一种或两种以上的混合物,

Q为B、W、P、V中的一种或两种以上的混合物,

 $a = 0.1 \sim 6$, b、c、d、e、g = 0.1 ~ 10 , f = > 0 ~ 0.5 , x 为满足其它元素化合价所需的氧原子总数。

催化剂所用的载体为二氧化硅或氧化铝或两者的混合物,其含量为催化剂总量的(40~60)%(w.t.)。

然而,X最好为Mg或Co或其混合物;Y最好为La或Ce或其混合物;Z最好为Rb或Cs或其混合物;Q最好为W或V或其混合物。载体最好选用二氧化硅。

催化剂可用本领域技术人员熟悉的混浆法制备,或用通常熟知的其它方法进行制备,用于制备催化剂各组份的原料一般采用硝酸盐或其它可溶于水的盐类或酸类,以便各组份混合均匀。

与已有技术相比,本发明提供的催化剂具有较好的氧化还原能力, 在范围更宽的反应条件下有很好的氧化还原稳定性,从而可以较低的空 气/烯烃比以及较低的氨/烯烃而仍能得到较高的产物单收,在保持较 高的烯烃转化率的前提下,丙烯腈或甲基丙烯腈的选择性有较显著的提 高。另外,本发明提供的催化剂还相对具有更低的反应操作温度,这将



有利于提高催化剂的使用寿命。

下面将通过实施例来进一步描述本发明的细节,通过实施例的评价数据可明显地看出本发明的积极效果。

在以下给出的实施例中,对催化剂考察评价的具体条件为:

反应器 : 流化床反应器, 内径 38mm

催化剂填装量: 550 克

反应器顶压力: 0.085 MPa (表压)

反应温度 : 420/410 ℃

反应时间 : 4 小时

原料比(摩尔比): 丙烯/氨/空气=1/1.15或1.05/9.5

WWH : 0.06

反应产物用 0℃酸液和水吸收,用气相色谱和化学分析结合分析产物。并计算碳平衡,当碳平衡在(95~105)%时为有效数据。

丙烯转化率、丙烯腈收率和选择性的定义为:

丙烯转化率	(0/) _	反应消耗的丙烯摩尔数	- ×100	%
	(%) =	丙烯进料摩尔数	~ 100	
丙烯腈选择性	(0/)	生成丙烯腈的摩尔数	— × 100	%
	(%)=	反应消耗的丙烯摩尔数	× 100	
丙烯腈单收	(0 ()	生成丙烯腈的摩尔数	100	%
	(%) = -		- × 100	

实施例 1.

将 423.2 克 (NH₄) ₆Mo₇O₇₄· 4H₂O 加入到 330 克 70℃的温水中,搅拌 使其全部溶解,再加入 1250 克 40% (w. t.)的硅溶胶,制成物料 A。

将 166 克 Fe (NO₃)₃·9H₂O 加入 40 克的 70℃热水中, 搅拌溶解后再加入 91.3 克 Bi (NO₃)₃·5H₂O , 292.8 克 Ni (NO₃)₃·6H₂O , 104.5 克 Mg (NO₃)₃·6H₂O , 46.7 g Sm (NO₃)₃·nH₂O , 搅拌溶解后制成物料 B。

在 20 克水中加入 1.9 克 KNO,和 1.5 克 RbNO, 溶解后将其加入物料 B 中形成物料 C。

将物料 C 在快速搅拌下滴加于物料 A 中,于 70℃老化 3 小时后喷雾干燥,将所得的颗粒在回转焙烧炉中于 580℃下焙烧 3 小时,得成品催化剂。

实施例 2~13

催化剂的制备方法同实施例 1,仅采用相应不同的起始物料及其用量。

实施例 1~13 制备催化剂的起始物料及相应的用量列于表 1,各催化剂活性组份的组成见表 2。

成品催化剂按上述考察评价条件进行考察, 其结果见表 3.

表 1. (单位: 克)

								_ :					
实施 例 13	实施例 12	实施例 11	实施例 10	实施例 9	实施例8	实施例7	实施例 6	实施例 5	实施例 4	外涵 図 3	实施例 2	实施例1	
365.8	394.5	412.5	401.7	412.5	405.3	416.1	398.1	394.5	405.3	408.7	419.4	423.2	(NH4)6 M07024* 4H20
354.9	142.0	182.5	111.5	243.4	50.7	456.3	405.6	101.4	20.3	20.3	50.7	101.4	BI (NO3)3 •SH2O
166.0	149.4	199.2	149.4	207.5	265.6	149.4	207.5	157.7	132.8	199.2	124.5	149.4	Fe (NO3)3 •9H ₂ O
381.7	238.6	268.4	405.6	310.1	345.9	369.8	238.6	149.1	280.3	268.4	292.2	405.6	Ni (NO3)2 •6H2O
94.1	146.3	94.1	73.2	104.5	167.2	104.5	104.5	62.7	104.5	94.1	125.4	104.5	Mg (NO3)2 •6H2O
17.7						26.6		71.0		53.2	26.6		Ce (NO3)3 •6H2O
71.0					44.4	35.5	44.4		17.8			44.4	(NO3)3 •nH2O
													Sm (NO3)3 •nH2O
138.0	55.2		96.6	117.3	96.6	72.5	117.3	138.0	117.3	69.0	41.4	1	Sb203
11	20.3	11	10.2	11	4.1	4.1	11	11	10.2	11	11	11	Co 3
7.3	36.3			7.3	3.6		14.5						Mn (NO ₃)2
241.9	134.4	302.4	235.2	121.0									Pb (NO ₃₎₂
		1.5	0.8			1.7	0.8	0.4	1.1	0.4	1.7	1.9	KN03
0.6	4.5	1.5	2.7	2.7	2.4	1.5		2.4	0.6	2.1		2.4	RbN03
3.2		1		2.4	2.0		2.0	2.0	1.6	2.4	0.4		CaNO3
	1.2	0.4				0.8	0.4	1			0.9		CONT
49.0	9.8	14.7	39.2		14.7		19.6	39.2		4.9	14.7		H ₇ P (W ₂ O ₇) •nH ₂ O
18.8	18.8	4.7		11.8	9.4	9.4	11.8	4.7	16.5	11.8		4.7	og NH



实施例 1.	$K_{0.09}Mo_{11.8}Bi_{1}Fe_{1.8}Ni_{6.8}Mg_{2}La_{0.5}Rb_{0.05}V_{0.2}$
实施例 2.	$K_{0.08}Mo_{11.7}Bi_{0.5}Fe_{1.5}Ni_{4.9}Mg_{2.4}Sb_{1.2}Ce_{0.3}Na_{0.05}Cs_{0.01}W_{0.3}\\$
实施例 3.	$K_{0.02}Mo_{11.4}Bi_{0.2}Fe_{2.4}Ni_{4.5}Mg_{1.8}Sb_{2}Ce_{0.6}Rb_{0.07}Cs_{0.06}V_{0.5}W_{0.1}$
实施例 4.	$K_{0.05}Mo_{11.3}Bi_{0.2}Fe_{1.6}Ni_{4.7}Mg_{2}Sb_{3.4}Cr_{0.5}La_{0.2}Rb_{0.02}Cs_{0.04}V_{0.7}\\$
实施例 5.	$K_{0.02}Mo_{11}Bi_{1}Fe_{1.9}Ni_{2.5}Mg_{1.2}Sb_{4.0}Ce_{0.8}Rb_{0.08}Cs_{0.05}V_{0.2}W_{0.8}$
实施例 6.	$K_{0.04}Mo_{11.1}Bi_{4.0}Fe_{2.5}Ni_{4.0}Mg_{2}Sb_{3.4}La_{0.5}Mn_{0.4}Cs_{0.05}Na_{0.02}V_{0.5}W_{0.4}\\$
实施例 7.	$K_{0.08}Mo_{11.6}Bi_{4.5}Fe_{1.8}Ni_{6.2}Mg_{2}Sb_{2.1}Ce_{0.3}La_{0.4}Cr_{0.2}Rb_{0.05}Na_{0.04}V_{0.4}\\$
实施例 8.	$Mo_{11.3}Bi_{0.5}Fe_{3.2}Ni_{5.8}Mg_{3.2}Sb_{2.8}La_{0.5}Cr_{0.2}Mn_{0.1}Rb_{0.08}Cs_{0.05}V_{0.4}W_{0.3}$
实施例 9.	$Mo_{11.5}Bi_{2.4}Fe_{2.5}Ni_{5.2}Mg_2Sb_{3.4}Pb_{1.8}Mn_{0.2}Rb_{0.09}Cs_{0.06}V_{0.5}$
实施例 10.	$K_{0.04}Mo_{11.2}Bi_{1.1}Fe_{1.8}Ni_{6.8}Mg_{1.4}Sb_{2.8}Pb_{3.5}Cr_{0.5}Rb_{0.09}W_{0.8}$
实施例 11.K _{0.}	$_{07}Mo_{11.5}Bi_{1.8} Fe_{2.4}Ni_{4.5}Mg_{1.8}Cu_{2}Pb_{4.5}Rb_{0.05}Na_{0.02}V_{0.2}W_{0.3}$
实施例 12.Mc	$b_{11}Bi_{1.4}Fe_{1.8}Ni_{4.0}Mg_{2.8}Sb_{1.6}Pb_{2}Mn_{1}Cr_{1}Rb_{0.15}Na_{0.06}V_{0.8}W_{0.2}$
实施例 13	$Mo_{10.2}Bi_{3.5}Fe_2Ni_{6.4}Mg_{1.8}Sb_4Pb_{3.6}Ce_{0.2}La_{0.8}Mn_{0.2}Rb_{0.2}Cs_{0.08}V_{0.8}W_1$

注: 上述催化剂的载体 SiO, 为 50% (w.t.)

表 3.

	丙烯腈单收	丙烯转化率	丙烯腈选择性	反应温度	氨/丙烯	
	(%)	(%)	(%)	(3)		
实施例 1	84.31	98.81	85.33	420	1.15	
	83.75	99.42	84.24	410	1.05	
实施例 2	83.68	98.78	84.71	420	1.15	
实施例3	80.43	98.84	81.37	420	1.15	
实施例 4	83.57	98.48	84.86	420	1.15	
实施例 5	84.72	98.93	85.64	420	1.15	
	84.04	99.66	84.33	410	1.05	
实施例 6	84.80	98.97	85.68	420	1.15	
	83.68	99.71	83.92	410	1.05	
实施例 7	81.78	98.42	83.09 •	420	1.15	
实施例 8	84.95	98.78	86.00	420	1.15	
	84.15	99.59	84.50	410	1.05	
实施例 9	83.45	98.82	84.45	420	1.15	
实施例 10	84.82	98.96	85.71	420	1.15	
	83.96	99.82	84.11	410 .	1.05	
实施例 11	82.74	98.81	83.74	420	1.15	
实施例 12	84.59	98.74	85.67	420	1.15	
	83.41	99.58	83.67	410	1.05	
实施例 13	85.18	98.86	86.16	420	1.15	
	84.78	99.46	85.24	410	1.05	